

Prediction of Super Critical Oil Extraction Yield Using Single and Combined Intelligent Systems

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Abstract

Simulation of Supercritical Fluid Extraction (SFE) of oil from pomegranate seeds using Supercritical Carbon Dioxide (SC-CO₂) was investigated to study the influence of process parameters on the extraction rate and oil composition. Yield predictions of the pomegranate seed oil production outbreaks from input vectors of our experiments may help us to interpret well to find optimized operating condition to achieve high yields. Intelligent systems are perfect tools for prediction in such systems with numerous effective factors that each one can change the experimental output answers maybe against expect. Our effort in this paper is to find a route and effective way to evaluate and predict the yield of seed oil production at different operational conditions without paying extra costs and spending more times by using intelligent systems. Several approaches to predict the pomegranate seed oil extraction with fuzzy sets; neural and adaptive fuzzy neural systems are analyzed and tested. Prediction strategies tested in the paper include the fuzzy C-means (FCM) clustering, the common neural networks (NN) and application of fuzzy neural networks. The results indicate the superiority of the adaptive fuzzy neural networks method over common neural network and fuzzy clustering approaches. The experimental results demonstrate that the proposed fuzzy neural network algorithm is able to reveal a better performance than conventional back propagation NN and FCM algorithms.

Keywords

Pomegranate Seed Oil; Adaptive Fuzzy Neural Networks; Fuzzy Clustering; Back Propagation

Introduction

Supercritical Fluid Extraction

Extraction of compounds from natural sources is the most widely studied application of supercritical fluids (SCF) with several of published scientific papers (Asis et al., 2006; Beis & Dunford, 2006; Bozan & Temelli, 2002; Bulley, Fattori, Meisen, & Moyls, 1984; J. D. Valle & Fuente, 2006; J. M. D. Valle & Uquiche, 2002).

Supercritical fluid extraction has advantages over traditional extraction techniques: it is a flexible process due to the possibility of continuous modulation of the solvent power/selectivity of the SCF, allows the elimination of polluting organic solvents and of the expensive post-processing of the extracts for solvent elimination (Salgin, Calimli, & Uysal, 2004). The influence of the main operating conditions of extraction, namely, the temperature and pressure of extraction on the oil extraction yield was performed.

Pomegranate seed oil consists of 65–85% conjugated fatty acids, the most important of which is 9-trans, 11-cis, 13-trans, Octadecatrienoic acid, the so-called Punicic acid. Pomegranate seed oil is reported to inhibit the upstream eicosanoid enzyme Phospholipase A₂ expressed by human prostate cancer cells (Salgin, Calimli, & Uysal, 2004; Schubert, Lansky, & Neeman, 1999). Supercritical fluid extraction has advantages over traditional extraction techniques: it is a flexible process due to the possibility of continuous modulation of the solvent power/selectivity of the SCF, allows the elimination of polluting organic solvents and of the expensive post-processing of the extracts for solvent elimination.

Artificial Neural Network (ANN)

Artificial neural networks (ANNs) have been generated considerable interest in the diverse fields of engineering as problem solving tools. It provides non-linear mapping between inputs and outputs. For this purpose, each input is multiplied by a weight, the inputs are summed and this quantity is operated on by the transfer function of the neuron to generate the output. The Multilayer Perceptron (MLP) network is probably the most often considered member of the neural network family, Fig. 1. The main reason for this is its ability to model simple as well as very complex functional relationships. This has been proven through

a large number of practical applications (Coulibaly & Baldwin, 2005; Hornik, Stinchcombe, & White, 1989; Mandal, Sivaprasad, & Dube, 2007; Sargolzaei, Asl, & Moghaddam, 2012; Sargolzaei & Kianfar, 2009; Sargolzaei, Saghatroleslami, Khoshnoodi, & Mosavi, 2006).

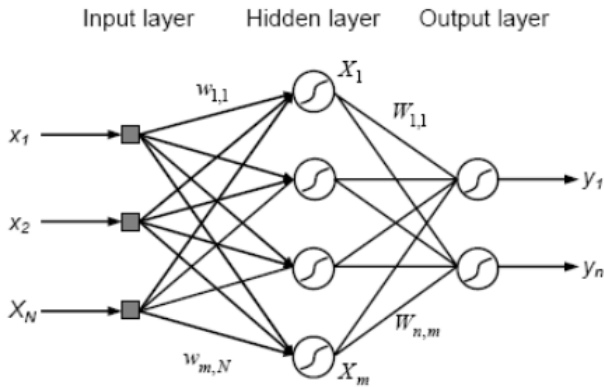


FIG. 1 A MULTI-LAYER NEURAL NETWORK WITH ONE HIDDEN LAYER OF NEURON

The outputs of MLP-networks is described as follow by considering the activation functions (f, F), based on hyperbolic tangent and linear functions :

$$\hat{y}_i(w, W) = F_i \left(\sum_{j=1}^q W_{ij} h_j(w) + W_{i0} \right) = F_i \left(\sum_{j=0}^q W_{ij} f_j \left(\sum_{l=1}^m w_{jl} z_l + w_{j0} \right) + W_{i0} \right) \quad (1)$$

The weights (specified by the matrices w and W) are the adjustable parameters of the network. The training data are a set of inputs ($u(t)$) and corresponding desired outputs ($y(t)$). Specify the training set by:

$$Z^N = \{[u(t), y(t)] \mid t = 1, \dots, N\} \quad (2)$$

The objective of training is determining a mapping from the set of training data to the set of possible weights to produce the network outputs $\hat{y}(t)$ close to the true outputs $y(t)$.

$$Z^N \rightarrow \hat{\theta} \quad (3)$$

The prediction error approach, based on the introduction of a measure of closeness in terms of a mean square error criterion is described as follow:

$$V_N(\theta, Z^N) = \frac{1}{2N} \sum_{t=1}^N (y(t) - \hat{y}(t|\theta))^T (y(t) - \hat{y}(t|\theta)) \quad (4)$$

The weights are then found as:

$$\hat{\theta} = \arg \min_{\theta} V_N(\theta, Z^N) \quad (5)$$

And iterative minimization scheme for weight learning based on learning factor is considered as:

$$\theta^{(i+1)} = \theta^{(i)} + \mu^{(i)} f^{(i)} \quad (6)$$

θ^i Specifies the current iterate (number 'i'), f^i is the search direction, and μ^i the step size.

Fuzzy C-Means Clustering (FCM)

Fuzzy c-means (FCM) is a data clustering technique wherein each data point belongs to a cluster to some degree that is specified by a membership grade. This technique was investigated as an improvement on earlier clustering methods. It provides a method that shows how to group data points that populate some multidimensional space into a specific number of different clusters (Khan, Anjum, & Bada, 2010; Tipsuwanporn, Intajag, Witheephanich, Koetsam-ang, & Samiamag, 2004; Tsoukalas & Uhrig, 1997).

The goal of traditional clustering is to assign each data point to one and only one cluster. In contrast, fuzzy clustering assigns different degrees of membership to each point. The membership of a point is thus shared among various clusters. This creates the concept of fuzzy boundaries which differs from the traditional concept of well-defined boundaries.

This procedure asserts that the well-defined boundary model usually does not reflect the description of real data. This assertion led him to develop a new family of clustering algorithms based on a fuzzy extension of the least-square error criterion. Typical of parametric approaches, this algorithm attempts to minimize a cost-function and a local minimizer is attained, instead of a global. In this case the following cost-function is minimized, with respect to U , a fuzzy K -partition of the data set, and to C , a set of K prototypes (cluster centers):

$$J_q(U, C) = \sum_{j=1}^m \sum_{i=1}^K (u_{ij})^q d^2(X_j, C_i); K \leq N \quad (7)$$

where q is any real number greater than 1, X_j is the j th n -dimensional feature vector, C_i is the centroid of the i th cluster, u_{ij} is the degree of membership of X_j in the i th cluster, $d^2(X_j; C_i)$ is any inner product metric (distance between X_j and C_i) M is the number of data

points, K is the number of clusters. The parameter q is the weighting exponent for u_{ij} and controls the “fuzziness” of the resulting clusters.

Adaptive Fuzzy Neural Networks

The fuzzy neural network implemented in this work can be represented by one hidden layer feed-forward architecture with N input units, K hidden units and M output ones which illustrated in Fig. 2 (Denai, Palis, & Zeghib, 2004).

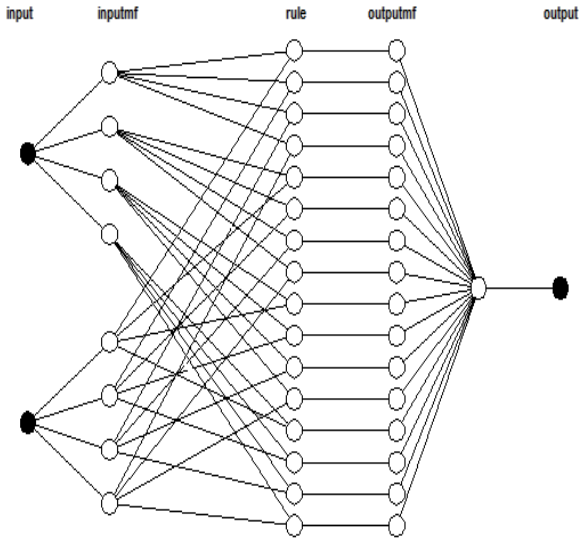


FIG. 2 THE ARCHITECTURE OF FUZZY-NEURAL NETWORK

Each input neuron represents a crisp input value. Each connection between input and hidden units has a weight equal to 1. Each unit in the hidden layer represents a fuzzy set over the input space \mathcal{R}^N . The output value of the i th hidden neuron for a given input vector $X = [x_1, x_2, \dots, x_N]$ can be interpreted as the degree of membership of X to the fuzzy set represented by this neuron. Neurons in the hidden layer are fully connected with neurons in the output layer. Information of the membership degree of vector X to each fuzzy set is aggregated in the output layer. Assuming the Gaussian form of the membership function in the fuzzy sets represented by hidden neurons, the output of the i th hidden neuron given X as input is equal to:

$$\mu_i = \exp \left\{ - \left(\frac{\|X - C_i\|}{\sigma_i} \right)^2 \right\} \quad (8)$$

Where $\| \cdot \|$ is the Euclidian norm, $\sigma_i \in \mathcal{R}, C_i = [c_{i1}, c_{i2}, \dots, c_{iN}]^T \in \mathcal{R}^N$ are parameters associated with a given neuron. Denoting by $W = [w_{km}]^{K \times M}$ the weight matrix of connections between

the hidden layer and the output layer, the input value for the j th output neuron is equal to:

$$u_j = \sum_{k=1}^K w_{kj} \mu_k \quad (9)$$

If we denote

$$s = \sum_{k=1}^K \mu_k \quad (10)$$

then the output value of that neuron is defined as:

$$y_j = \frac{u_j}{s} = \frac{\sum_{k=1}^K w_{kj} \mu_k}{\sum_{k=1}^K \mu_k} \quad (11)$$

The FNN model described here can be interpreted in terms of an equivalent fuzzy system. For the i th neuron in the input layer (the fuzzification neuron), a fuzzy IF-THEN rule R_i can be extracted:

$$R_i : \text{IF } X \text{ is } \mu_i \text{ THEN } y_1 = W_{i1} \text{ AND } \dots \text{ AND } y_M = W_{iM} \quad (12)$$

It is clear that fuzzy properties μ_i as well as the (crisp) outputs w_{ij} of the fuzzy rules are determined in the training process. Certainly the notion of μ_i indicates only a fuzzy set over the input space, not any linguistic value. The fuzzy rule set is then defined as:

$$S = \{R_i : i = 1, K\} \quad (13)$$

Materials and Methods

Materials

Squalane (99% pure, Sigma-Aldrich, USA), hexane and methanol (HPLC grade, Sigma-Aldrich, USA), ethanol (PA grade, Panreac Quimica, Spain), and a FAME mixture C8–C24 (PA grade, Supelco, Bellefonte, USA) were purchased and used as received. Acetyl chloride (PA grade) was purchased from Merck (Germany) and redistilled before use. Carbon dioxide was supplied with a purity of 99.99% from Khorakian Co. (Iran).

Sample Preparation

The pomegranate selected for this study obtained from Saveh in the Markazi province of Iran. After transferring the fruit to the laboratory, those with the defective parts (i.e., those with sunburns, cracks, and bruises in the husk) were discarded. Then, seeds from the rest of the fruit were separated from the juice and washed carefully to remove sugars and other adhering

materials. Separated seeds were placed in an oven (Indelab, Model 6882A, Malaysia) at 313K until a constant weight was reached. Dried pomegranate seeds pulverized in a food grinder (Hanil Science Industrial Co. Ltd., Korea) to a particle size distribution less than 40-mesh as measured by a sieve (Chung Gye Sang Gong Sa, Korea) was used for the extraction.

SFE experiments were carried out in the extraction apparatus shown schematically in Fig. 3. Gaseous CO₂ taken from a cylinder, is first compressed to the desired extraction pressure by means of a gas compressor, Nova Swiss (Model 5542121), and then heated to the desired temperature by passing through a high pressure tubing coil immersed in a temperature controlled (up to ± 0.2 K) water heating bath. SC-CO₂ then flows at the desired pressure and temperature conditions upwards through a packed bed of pomegranate seeds contained in the extraction vessel (316 SS(L); internal diameter of 20mm; total length of 712 mm). To avoid undesired entrainment effects the pomegranate seeds packed bed is placed in the extraction vessel between two metallic porous plates and the extra space filled with cotton. The extractor is heated by passing hot water through a heating jacket surrounding the outer surface of the vessel. The extraction pressure was controlled by means of a back pressure regulator valve, BPR (Tescom 27-1700, JAPAN) where depressurization of CO₂ flow stream exiting the extraction vessel took place. The extracted substances were precipitated and collected into a glass trap, immersed in an ice bath. To ensure a total recovery of compounds, the gas flow coming out from the first trap passes through a second glass trap, filled with n-hexane. The gas flow rate and total mass of CO₂ used in the experiment are measured with a Coriolis type gas flow meter (Danfoss, Model Mass 6000, UK). The extraction pressure is measured at the entrance of the extraction vessel with an accuracy of ± 0.1 MPa (Wika, Model 881.14.600, UK).

SFE of oil from pomegranate seeds was carried out in the pressure range of 20.0-40.0MPa and in the temperature range of 313–333 K. An additional extraction run was performed with the addition of ethanol as co-solvent. This experiment was carried out at 323K and 20.0MPa of temperature and pressure, respectively, with a co-solvent mass flow rate of 0.7 gmin⁻¹, corresponding to ethanol: CO₂ mass ratio of 6.5:93.5 (w/w). In this experiment only three extract samples were taken, at 50, 120 and 180 min.

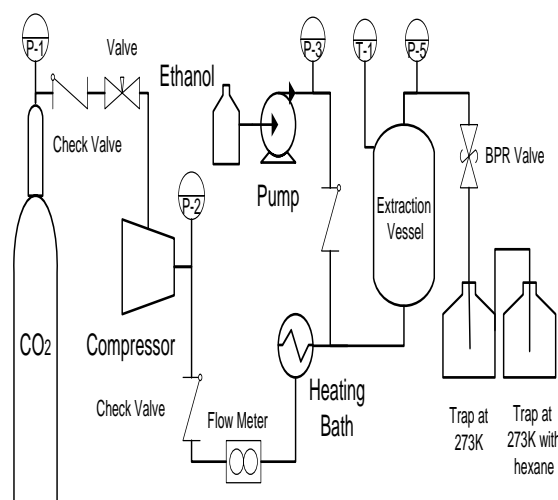


FIG. 3 HIGH PRESSURE APPARATUS FOR SUPERCRITICAL FLUID EXTRACTION OF POMEGRANTE SEED OIL

Table 1 shows the experimental conditions at which the SFE experiments were carried out as well as the oil extraction yield obtained for each experiment (expressed as %, goil/100 gdry pomegranate seeds). SC-CO₂ was able to extract, at maximum, ca. 14% of the total oil contained in the raw material after 3hr of extraction (extraction relative to Soxhlet extraction results).

TABLE 1 EXPERIMENTAL CONDITIONS OF THE EXTRACTION EXPERIMENTS

Data set	P (MPa)	T(K)	Yield g oil/100 g dry pomegranate seeds
1	20.0	313	0.90 \pm 0.1 ^a
2	20.0	323	0.88 \pm 0.1 ^a
3	20.0	333	0.85 \pm 0.2 ^a
4	30.0	313	1.97 \pm 0.1 ^a
5	30.0	323	1.93 \pm 0.2 ^a
6	30.0	333	1.88 \pm 0.1 ^a
7	40.0	313	2.66 \pm 0.1 ^a
8	40.0	323	2.58 \pm 0.2 ^a
9	40.0	333	2.46 \pm 0.1 ^a
10 ^c	20.0	323	17.30 \pm 0.6 ^b

a Mean values \pm standard deviation (n = 2). Values in a row followed by different letters (a-b) are significantly different (P < 0.05).

c This run was carried out with ethanol as a co-solvent, at a ethanol:CO₂ mass ratio of 6.5:93.5 (w/w).es

Seeds oil extraction yields increase significantly (P<0.05) with pressure at constant temperature, which follows the usual trend for the SFE of lipids from food residues (Bulley, Fattori, Meisen, & Moyls, 1984). This increase is explained by the increased CO₂ density with the pressure, and thus its solvent capacity to solubilize the lipids from the pomegranate seeds (Poling, Prausnitz, & O'Connell, 2001; Reverchon & Marco, 2006). The oil extraction yield obtained with the addition of ethanol as co-solvent (6.5 wt%) was

significantly ($P < 0.05$) higher than the one obtained with pure SC-CO₂ at the same process conditions of 323K and 20.0 MPa. This increase in the oil extraction yield of pomegranate seeds with the addition of an alcohol as co-solvent is in agreement with that observed by other authors. It can be attributed to an increase in the solvent density and/or to modifications in the physical and chemical intermolecular forces in the system (Abbasi, Rezaei, & Rashidi, 2008; Guclu-Ustundag & Temelli, 2005; Ju et al., 2010; Li, Wu, Rempel, & Thiyam, 2010; Sun, Xu, Saldana, & Temelli, 2008).

Our effort in this paper is to find a route and effective way to evaluate and predict the yield of seed oil production at different operational conditions without paying extra costs and spending more times by using intelligent systems. Finding and comparing some practical simulations and optimizing the answers and presenting the best way have been done in this paper.

Result and Discussion

In order to evaluate the performance of the three proposed algorithms for prediction purposes, integral absolute error (IAE) and mean square error (MSE) are considered as two common statistical measures. The comparative performances of the adaptive fuzzy neural network approach are evaluated respect to back propagation neural network (NN) and fuzzy C-Means clustering algorithm on a similar set of pomegranate seed oil data captured from our previous work. The optimal neural network topology is obtained by considering the different number of hidden layers for training of the neural network. Table 2 demonstrates that our training algorithm is optimized by selecting 7 neurons in hidden layer and it shows that obtained results in terms of IAE and MSE are minimized by selecting 3 hidden layers. Also the corresponding statistical efficiency coefficients (R^2) have been illustrated from the neural network algorithm in Tables 2 and 3. Generally speaking R value greater than 0.9 indicates a very satisfactory model performance, while R value in the range 0.8– 0.9 signifies a good performance and value less than 0.8 indicate an unsatisfactory model performance. So, the structure of the utilized NN has been configured with 2 input neurons, 7 hidden neurons in each of three hidden layers and one output neuron. Simulation of the pomegranate seed oil production yield on the neural network with proposed structure is illustrated in Fig 4.

TABLE 2 EVALUATION CHARACTERS FOR ANN STRUCTURE WITH 7 NEURONS AND DIFFERENT HIDDEN LAYERS

Number of Hidden Layers with 7 Neurons	Neural Network		
	IAE	MSE	R ²
1	8.25	0.73156	0.56
2	5.106	0.3826	0.772
3	3.615	0.169	0.9193
4	3.7135	0.1863	0.9124
5	3.9913	0.2078	0.9154
6	6.6792	0.6119	0.7066
7	4.4646	0.2687	0.8653
8	5.936	0.411	0.8079
9	6.5041	0.5554	0.696
10	4.7194	0.3125	0.8353

TABLE 3 THE RESULTS OF FUZZY C-MEANS CLUSTERING ALGORITHMS

Radius	MSE Train	MSE test
0.5	0.0494	38.2355
0.4	4.3615e-014	269.6974
0.3	6.2911e-016	10.0062
0.2	5.1872e-016	0.4292
0.1	3.5255e-016	0.4704
0.08	2.5384e-016	0.4685
0.06	2.3802e-016	0.4637
0.04	2.3489e-016	0.4616
0.02	1.6847e-016	0.4615

TABLE 4 DATA ESTIMATION AND PREDICTION PERFORMANCES DUE TO ADAPTIVE FUZZY NEURAL NETWORK ALGORITHM

Number of Rules	MSE for Training Data	MSE for Testing Data
2	0.29	0.38
3	0.0312	0.356
4	0.0217	0.0837
5	9.057e-6	0.31
6	9.65e-6	0.272
7	2.13e-5	0.109
8	7.26e-6	2.96
9	1.73e-5	0.873
10	1.91e-5	1.22

TABLE 3 THE STRUCTURES OF BACK PROPAGATION IN NEURAL NETWORK SIMULATIONS FOR 3 HIDDEN LAYERS

no.	no. of neurons in each layer			The best no. of neurons in each layer			Calculated performance for the best structure		
		Layer 1		Layer 2	Layer 3		SSE	MAE	R ²
1	1:10								
	1:10	9		8	1		2.559177e+000	3.850170e-001	8.8746e-001
	1								
2	1:2:10								
	1:2:10	7		9	5		3.436280e-001	1.484096e-001	9.7380e-001
	1:10								
3	1:2:20								
	1:2:20	11		11	5		2.366016e-001	1.245456e-001	9.8460e-001
	5								
4	8:13								
	8:13	11		10	7		7.158939e-001	2.136374e-001	9.6013e-001
	1:10								
5	11								
	11	11		11	18		3.057050e+000	2.952519e-001	7.2673e-001
	1:50								
6	1:20								
	1:20	20		11			4.876722e-001	1.549958e-001	9.6224e-001
7	1:40								
	1:40	15		18	13		6.826126e-001	1.927563e-001	9.4466e-001
	1:40								
8	10:12								
	10:12	10		10	5		2.372642e-001	1.199101e-001	9.8742e-001
	5								

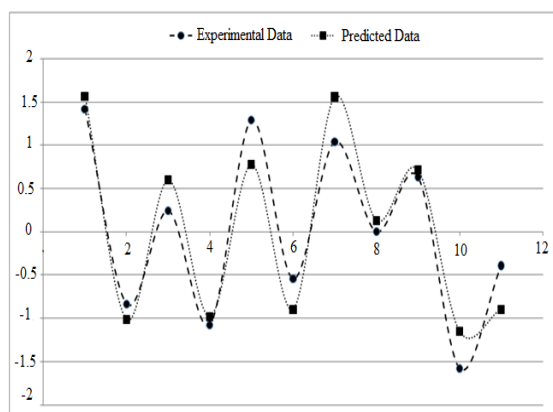


FIG. 4 EXPERIMENTAL AND SIMULATED SEED OIL EXTRACTION DATA ALONG WITH ANN STRUCTURE WITH 7 NEURONS AND DIFFERENT HIDDEN LAYERS (ITEM 3 FROM TABLE 2)

Our results have been developed by trying on other back propagation architectures and the results showed that for 3 hidden layers better approaches were achievable. Table 3 shows some of the tried simulations among the 1000 simulations which have been run according to written code in Matlab 2008.

Fig. 5 shows different structures of the neural network with 3 hidden layers. In each method neural network is trained 3 times and the values of IAE and MSE are computed in each time for reliability consideration, so their averages are considered for comparing.

The results of fuzzy C-Means clustering algorithms have been summarized in Table 4. Fig.6 illustrates the comparative results corresponding to estimation of pomegranate seed oil by the FCM algorithm. As shown, FCM algorithm is able to predict the pomegranate seed oil parameter with a better performance compared to the NN algorithm.

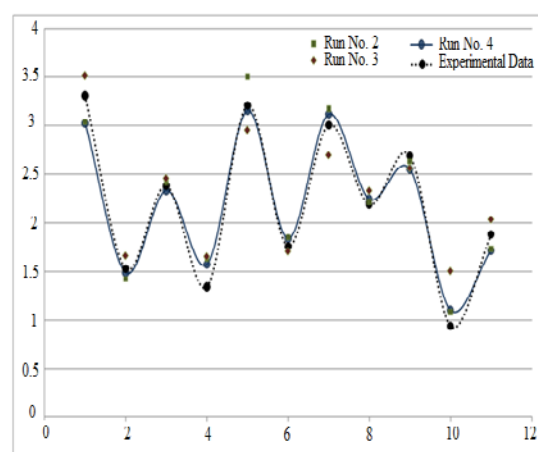


FIG. 5 COMPARISON PROFILES OF EXPERIMENTAL POMEGRANTE SEED OIL EXTRACTION AND OUTPUT OF DIFFERENT ANN SIMULATION STRUCTURE (DATA ARE AVAILABLE FROM TABLE 3)

Data estimation and prediction performances due to adaptive fuzzy neural network algorithm have been summarized in Table 5 and Fig. 7. Table 5 consist of the MSE for the both training and testing data.

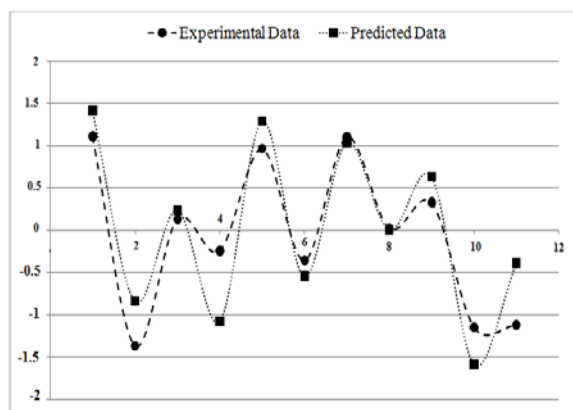


FIG. 6 COMPARISON PROFILES OF EXPERIMENTAL POMEGRANTE SEED OIL EXTRACTION AND OUTPUT OF FUZZY C-MEANS CLUSTERING ALGORITHMS (DATA ARE AVAILABLE FROM TABLE 4 WITH RADIUS=0.2)

Table 5 and Fig 7 indicate that the proposed adaptive fuzzy neural network methodology could illustrate a very good performance than other approaches. It shows that the proposed algorithms outperform the BP neural network and fuzzy C-Means clustering algorithm in accuracy and reliability. So, the minimum MSE and IAE measures are obtained by adaptive fuzzy neural network algorithm with much better performance in parameter prediction than FCM and NN algorithms.

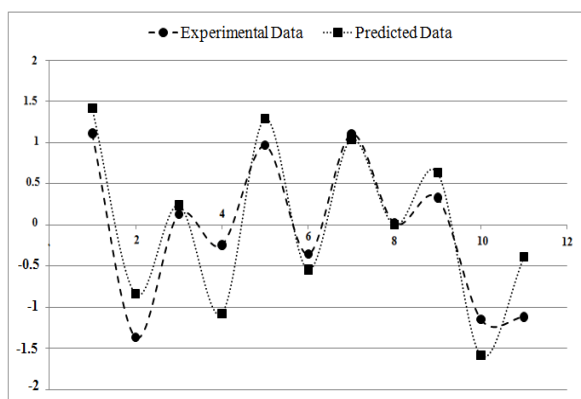


FIG. 7 COMPARISON PROFILES OF EXPERIMENTAL POMEGRANTE SEED OIL EXTRACTION AND OUTPUT OF ADAPTIVE FUZZY NEURAL NETWORK ALGORITHM (DATA ARE AVAILABLE FROM TABLE 5, NUMBER OF RULES=4)

CONCLUSIONS

Our effort in this paper is to find a route and effective way to evaluate and predict the yield of seed oil production at different operational conditions without paying extra costs and spending more times by using intelligent systems. In this paper several approaches to prediction of pomegranate seed oil with fuzzy sets; neural and adaptive fuzzy neural systems are analyzed and tested. Prediction strategies tested in the

paper include the fuzzy C-means (FCM) clustering, the common neural networks (NN) and application of fuzzy neural networks. The results indicate the superiority of the adaptive fuzzy neural networks method over common neural network and fuzzy clustering approaches. The experimental results demonstrate that the proposed fuzzy neural network algorithm is able to reveal a better performance than conventional back propagation NN and FCM algorithms.

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